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IS 1559 ( Part 1 ) : 1989

( Reaffirmed 1999 )

*Indian Standard*

# METHODS OF CHEMICAL ANALYSIS OF FERRO-SILICON

## PART 1 DETERMINATION OF SILICON

*( Second Revision )*

**भारतीय मानक**

**फेरो सिलिकोन के रासायनिक विश्लेषण की पद्धति**

**भाग 1 सिलिकॉन के ज्ञात करना**

**( दूसरा पुनरीक्षण )**

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

*January* 1990

**Price Group 1**

Methods of Chemicals Analysis of Ferrous Metals Sectional Committee, SMDC 2

## FOREWORD

This Indian Standard (Part 1) (Second Revision) was adopted by the Bureau of Indian Standards on 17 October 1988, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

This revision of this standard (Part 1) was published in 1982. This standard has been revised again on the basis of its implementation by testing laboratories and manufacturers. In this revision, the gravimetric method for determination of silicon has been modified in order to bring it in line with the international practices. This has been done by deriving assistance from ISO 4158 : 1978 'Ferro silicon, determination of silicon content — Gravimetric method', issued by the International Organization for Standardization (ISO).

This standard has been published in seven parts. This part covers the method for determination of silicon. The other parts are as follows:

Part 2 Determination of carbon

Part 3 Determination of sulphur

Part 4 Determination of phosphorus

Part 5 Determination of aluminium

Part 6 Determination of calcium

Part 7 Determination of manganese

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

# *Indian Standard*

# METHODS OF CHEMICAL ANALYSIS OF FERRO-SILICON

## PART 1 DETERMINATION OF SILICON

## *( Second Revision )*

## 1 SCOPE

This standard ( Part 1 ) covers the method for determination of silicon in the range of 15 to 85 percent in ferro-silicon.

## 2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

| *IS No.* | *Title* |
|---|---|
| IS 265 : 1976 | Specification for hydrochloric acid |
| IS 1472 : 1977 | Methods of sampling ferro-alloys for determination of chemical composition |

## 3 SAMPLING

The samples shall be drawn and prepared in accordance with IS 1472 : 1977.

## 4 DETERMINATION OF SILICON BY GRAVIMETRIC METHOD

### 4.1 Outline of the Method

After decomposition of the sample by fusion with sodium peroxide and sodium carbonate, silica is separated by dehydration with perchloric acid, ignition and weighing followed by hydrofluorization.

### 4.2 Reagents

**4.2.1** *Fusion Mixture,* sodium carbonate and sodium peroxide mixed in the ratio of 3 : 1.

**4.2.2** *Dilute Hydrochloric Acid,* ( 1 : 1 ) ( *v/v* ), 2 percent and 10 percent.

**4.2.3** *Concentrated Hydrochloric Acid,* ( rd = 1·16 ), conforming to IS 265 : 1976.

**4.2.4** *Perchloric Acid,* 70 percent.

**4.2.5** *Silver Nitrate,* 1 percent ( *m/v* ).

**4.2.6** *Hydrofluoric Acid,* 40 percent.

**4.2.7** *Dilute Sulphuric Acid,* 1 : 4 ( *v/v* ).

### 4.3 Procedure

**4.3.1** Weigh 0·25 g of finely ground sample, preferably 150 microns into a 25 to 30 ml nickel crucible ( silicon free iron crucible may be used ) containing 5 g of fusion mixture. Mix well and cover with 2 g of sodium peroxide. Cover the crucible and fuse the contents in a muffle furnace and heat at 400°C and maintain for 15 minutes. Alternatively, the fusion can be accomplished by holding the crucible with a pair of tongs and slowly revolving it around the outer edge of the flame until the contents have melted down, care being taken not to raise the temperature rapidly so as to cause spattering. When the mass is molten, a slight rotary motion is imparted to the crucible to stir up any unattacked particles of alloy on the bottom or sides of the crucible and contents being maintained at a low heat. Just before the completion of the fusion, which only requires three or four minutes, the temperature is increased to bright redness.

**4.3.2** Allow the crucible to cool, slowly transfer the crucible with its solidified melt in 50 ml of cold water in a 500 ml polythene beaker and cover the beaker immediately with a polythene cover plate. When the reaction has subsided, take out the crucible and lid and wash them thoroughly with warm hydrochloric acid ( 10 percent ) and then with water collecting all the washings in the same polythene beaker. Acidify the extract with 50 ml of concentrated hydrochloric acid, transfer the solution to a 500 ml glass beaker and add 60 ml of perchloric acid and place the beaker on a hot plate and heat until copious fumes of perchloric acid are evolved. Continue the operation of heating until the residue begins to crystallize and becomes pasty. Remove from hot plate, allow to cool and take it up in 20 ml of dilute hydrochloric acid ( 1 : 1 ) and few ml of hot water. Stir and dilute with 250 ml of boiling water. Mix well and allow to settle, filter the silica through a medium texture filter paper and wash with dilute hydrochloric acid ( 2 percent ) and finally several times with hot water till free from chloride.

NOTE — All traces of perchloric acid should be washed off, otherwise it may lead to explosion.

**4.3.3** Transfer the filtrate and washings to the beaker used for the initial dehydration. Evaporate to a volume about 250 ml, add 20 ml of perchloric acid and carry out second dehydration as described above. Cool and filter through a filter paper of medium texture and wash the precipitate as above but carrying out the final washings with cold water instead of hot water till free from chloride.

**4.3.4** Combine the two filter papers in a platinum crucible. Dry to remove excess of moisture and incinerate in the muffle furnace at a temperature of 1100°C to a constant mass; and cool and weigh.

**4.3.5** Moisten the residue with a few drops of water, add about 10 ml of hydrofluoric acid and 2 or 3 drops of dilute sulphuric acid. Heat it to dryness until sulphuric acid fumes just begins to evolve. Repeat second volatilization under the same conditions, using 2 ml hydrofluoric acid. Ignite in the muffle furnace at 1100°C to a constant mass. Allow to cool in a desiccator and weigh.

**4.3.6** *Blank*

Carry out blank on all reagents employed and correct the final results, if any.

**4.3.7 Calculation**

$$\text{Silicon, percent by mass} = \frac{(A - B) - C \times 46{\cdot}74}{D}$$

where

$A$ = mass in g of crucible before hydrofluoride treatment,

$B$ = mass in g of crucible after hydrofluorization,

$C$ = mass in g of silica from the blank, and

$D$ = mass in g of sample taken.

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